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(54) MAGNETIC THIN FILM AND FORMING METHOD THEREOF

(57)Abstract:

PROBLEM TO BE SOLVED: To obtain a magnetic thin film with high resistivity value and satisfactory magnetic property by setting a concentration of organic acid at a specific value in a plating bath relative to the concentration of Mo ions in a plating bath.

SOLUTION: A method for forming a magnetic thin film, whereby a plating bath containing Ni ions, Fe ions, Mo ions, and an organic acid is used for forming the magnetic thin film on a conductive film by an electroplating method, sets the concentration of the organic acid to about 3 to 20 times larger than that of the Mo ions in the plating bath. Then, for adding Mo ions into the plating bath, a substance such as sodium molybdate and ammonium molybdate is added to the plating bath. Furthermore, a salt of hydroxyl carboxylic acid is adopted as the organic acid added to the plating bath. A substance such as tartaric acid and lactic acid is adopted as the hydroxy carboxylic acid, and a substance such as sodium tartar, Rochelle salt, sodium lactate, and malate sodium is adopted as the salt of the hydroxy carboxylic acid. Moreover, the concentration of the organic acid added to the plating bath is set at 0.001 mol/L or more.

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CLAIMS

[Claim(s)]

[Claim 1] The formation method of the magnetic thin film characterized by being the formation method of the magnetic thin film which forms a magnetic thin film with electroplating on an electric conduction film, and the concentration of the aforementioned organic acid under aforementioned plating bath being 3 to 20 times the concentration of the aforementioned Mo ion under aforementioned plating bath using the plating bath containing nickel ion, Fe ion, Mo ion, and an organic acid.

[Claim 2] It is the formation method of the magnetic thin film characterized by the aforementioned organic acid being the salt of a hydroxy acid or a hydroxy acid in the formation method of a magnetic thin film according to claim 1.

[Claim 3] It is the formation method of the magnetic thin film characterized by the concentration of the aforementioned organic acid under aforementioned plating bath being [mol / 0.001 // 1. / or more] in the formation method of a magnetic thin film according to claim 1 or 2.

[Claim 4] It is the formation method of the magnetic thin film characterized by the concentration of the aforementioned organic acid under aforementioned plating bath being 0.005 or more mol/l in the formation method of a magnetic thin film according to claim 3.

[Claim 5] The magnetic thin film characterized by being the magnetic thin film formed by electroplating on the electric conduction film, forming the concentration of the aforementioned organic acid under aforementioned plating bath using the plating bath containing nickel ion, Fe ion, Mo ion, and an organic acid as three to 20 times of the concentration of the aforementioned Mo ion under aforementioned plating bath, and including 1 - 5atomic% Mo.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[The technical field to which invention belongs] this invention relates to a magnetic thin film and its formation method, and relates to the magnetic thin film which has high resistivity especially, and its formation method.

[0002]

[Description of the Prior Art] Conventionally, the magnetic thin film which was excellent in ***** is used as a magnetic core for thin film MAG devices, such as the thin film magnetic head, a thin film transformer, and a thin film inductor. Especially the magnetic thin film that consists of a permalloy (iron nickel alloy) has high permeability, is excellent in *****, and since a magnetostriction is also small, it is widely used for the up magnetic core of the recording head of a hard disk drive unit etc.

[0003] However, since the magnetic thin film which consists of a permalloy had resistivity as low as about 20 microohm-cm, it was difficult for loss by the eddy current to become large to RF magnetic field several 10MHz or more, and to acquire a good RF property. Then, the technology which forms the high magnetic thin film of resistivity is proposed by mixing Mo (molybdenum) in a permalloy.

[0004] For example, in JP,7-122426,A, the multilayer formed by forming a layer with few the high layers of resistivity and the amounts of Mo mixing with many amounts of Mo mixing one by one is indicated, and the purport this multilayer excels [purport] in ***** is indicated. This multilayer is formed by electroplating using the permalloy bath by which Mo ion was added, and the amount of Mo mixing in a magnetic thin film is adjusted by changing current density. In addition, the purport which adds a tartaric acid to a permalloy bath is indicated that the amount of Mo mixing in a magnetic thin film should be changed a lot.

[0005] Moreover, in JP,9-63016,A, the magnetic thin film which consists of the permalloy containing molybdenum, chromium, and a tungsten is indicated, and, as for this magnetic thin film, the purport below 1.0 oersteds (Oe) and whose resistivity coercive force Hc is 40 or more microohm-cm more than 1.5 tesla (T) for saturation magnetic flux density Bs is indicated.

[0006]

[Problem(s) to be Solved by the Invention] However, about how the amount of Mo mixing to the inside of a magnetic thin film is controlled proper, it was indicated at all by neither JP,7-122426,A nor JP,9-63016,A. Since the magnetic properties of the magnetic thin film which consists of a permalloy tend to change with the amounts of Mo mixing a lot, establishment of the technology which controls the amount of Mo mixing proper is very important.

[0007] The purpose of this invention has high resistivity and it is to offer the magnetic thin film which has good magnetic properties, and its formation method.

[0008]

[Means for Solving the Problem] The above-mentioned purpose is the formation method of the magnetic thin film which forms a magnetic thin film with electroplating on an electric conduction film using the plating bath containing nickel ion, Fe ion, Mo ion, and an organic acid, and it is attained by the formation method of the magnetic thin film characterized by the concentration of

the aforementioned organic acid under aforementioned plating bath being 3 to 20 times the concentration of the aforementioned Mo ion under aforementioned plating bath. Thereby, since the organic-acid concentration under plating bath to Mo ion concentration under plating bath is set as a suitable value, the amount of Mo mixing in a magnetic thin film can be set as a suitable value, and resistivity is high and can form the magnetic thin film which has good magnetic properties.

[0009] Moreover, as for the aforementioned organic acid, in the formation method of the above-mentioned magnetic thin film, it is desirable that it is the salt of a hydroxy acid or a hydroxy acid. Moreover, as for the concentration of the aforementioned organic acid under aforementioned plating bath, in the formation method of the above-mentioned magnetic thin film, it is desirable that it is [mol / 0.001 //l. / or more]. Moreover, as for the concentration of the aforementioned organic acid under aforementioned plating bath, in the formation method of the above-mentioned magnetic thin film, it is desirable that it is [mol / 0.005 //l. / or more]. Thereby, the amount of Mo mixing to the inside of a magnetic thin film can be set up stably.

[0010] Moreover, using the plating bath containing nickel ion, Fe ion, Mo ion, and an organic acid, the above-mentioned purpose is the magnetic thin film formed by electroplating on the electric conduction film, and the concentration of the aforementioned organic acid under aforementioned plating bath is formed as three to 20 times of the concentration of the aforementioned Mo ion under aforementioned plating bath, and it is attained by the magnetic thin film characterized by including 1 - 5atomic% Mo. Thereby, since 1 - 5atomic% Mo is contained in the magnetic thin film, resistivity is high and can offer the magnetic thin film which has good magnetic properties.

[0011] [Embodiments of the Invention] The formation method of the magnetic thin film by 1 operation form of this invention is explained using drawing 1 or drawing 7 . Drawing 1 is the schematic diagram showing the cell for electroplating. Drawing 2 is a graph which shows the relation between current density and the amount of Fe mixing to the inside of a magnetic thin film. Drawing 3 is a graph which shows the relation between Mo ion concentration and the amount of Mo mixing to the inside of a magnetic thin film. Drawing 4 is a graph which shows the relation between the value which *(ed) organic-acid concentration by Mo ion concentration, and the amount of Mo mixing to the inside of a magnetic thin film. Drawing 5 is a graph which shows the relation between the amount of Mo mixing to the inside of a magnetic thin film, and the resistivity of a magnetic thin film. Drawing 6 is a graph which shows the relation between the amount of Mo mixing to the inside of a magnetic thin film, and the saturation magnetic flux density Bs of a magnetic thin film. Drawing 7 is a graph which shows the relation between the amount of Mo mixing to the inside of a magnetic thin film, and the corrosion resistance of a magnetic thin film.

[0012] By the formation method of the magnetic thin film by this operation form forming a magnetic thin film with electroplating on an electric conduction film using the plating bath containing nickel ion, Fe ion, Mo ion, and an organic acid, and setting the organic-acid concentration under plating bath as about about 3 to 20 times of Mo ion concentration under plating bath, resistivity is high and it is characterized [main] by forming the magnetic thin film which has good magnetic properties.

[0013] First, how to form a magnetic thin film on an electric conduction film with electroplating is explained using drawing 1 . Drawing 1 is the schematic diagram showing the cell for electroplating. In drawing 1 , drawing on the right-hand side of space is drawing for the physical relationship of the hole of a shield and a cathode being shown, and is a thing at the time of seeing the cell shown in the space chart on the left from space right-hand side. As shown in drawing 1 , the shield 12 of plastics is formed in the container 10, and the circular hole 14 is formed in the shield 12. The disk-like anode 16 is formed in the space left-hand side in a container 10, the disk-like cathode 18 is formed in space right-hand side, and these anodes 16 and the cathode 18 have countered through the hole 14 formed in the shield 12. Moreover, the anode 16 is supported by the anode support fixture 20, and the cathode 18 is supported by the cathode support fixture 22.

[0014] For example, nickel board is used for an anode 16. Moreover, in drawing 1 , for

convenience, although the cathode 18 was illustrated in the same configuration as an anode, in manufacturing an actual device, the electric conduction film used as the ground for forming a magnetic thin film etc. will correspond to a cathode. nickel can use and Fe can use a 15 – 25atomic% binary alloy thin film etc. 75 – 85atomic% as this electric conduction film, for example. [0015] If the inside of a container 10 is filled with the plating bath 24, and an anode 16 is connected to the plus side of a power supply and it connects a cathode 18 to the minus side of a power supply, current will flow under a plating bath 24 through the hole 14 of a shield 12. Since the hole 14 which an anode 16 and a cathode 18 are disks-like, and was formed in the shield 12 is also circular, the current of almost uniform current density will flow for the plating bath 24 between an anode 16 and a cathode 18.

[0016] In this way, the magnetic thin film which consists of a permalloy will be formed in the front face of a cathode 18.

(Current density) The current density of the current which flows to the plating bath 24 between an anode 16 and a cathode 18 through the hole 14 formed in the shield 12 can be set as the range of 10–15mA/cm².

[0017] Drawing 2 measures the relation between current density and the amount of Fe mixing to the inside of a magnetic thin film to ask for suitable current density. The horizontal axis of drawing 2 shows the current density of the current which flows to the plating bath between an anode and a cathode, and the vertical axis shows the amount of Fe mixing to the inside of a magnetic thin film. In the range of 10 – 15 mA/cm², the amount of Fe mixing in a magnetic thin film has current density almost fixed as about 18 atomic(s)% so that drawing 2 may show. Therefore, if current density is set up in the range of 10 – 15 mA/cm², it will become possible to form the magnetic thin film by which composition was stabilized. In addition, current density is not necessarily limited to setting it as the range of 10 – 15 mA/cm², and it can be suitably set up so that the magnetic thin film of desired composition may be obtained.

[0018] (Plating bath) As a plating bath, the plating bath which can form a permalloy, for example, a Burns–Warner bath, a Wolf bath, a low concentration bath, etc. can be used. A Burns–Warner bath is a plating bath of the sulfate–chloride system proposed by R.M.Burns of a Bell lab, and C.W.Warner, and can form the magnetic thin film which consists of the permalloy of 80% of nickel mixing ****. plating conditions -- for example, a nickel sulfate -- 212 g/l and an iron sulfate (II) -- 25 g/l and a sodium sulfate can be made into 180 g/l, and temperature can be made [22 g/l and a nickel chloride / 18 g/l and ferric chloride (II)] into 50 degrees C for 2.5 g/l and a boric acid

[0019] A Wolf bath is a plating bath of the sulfate–chloride system which galvanizes in a magnetic field, in order to form the permalloy which has a magnetic anisotropy, and in order that it may decrease membrane stress, it is a plating bath by which saccharin is added (I. W.Wolf, J.Appl.Phys, 33, 1152, 1962). plating conditions -- for example, a nickel sulfate -- 293 g/l and an iron sulfate (II) -- 25 g/l and a concentrated sulfuric acid can be made into 14 ml/l, and saccharin can be made [6.2 g/l and a SAURIRU sodium sulfate / 0.42 g/l and a sodium chloride] into 0.5 g/l for 9.7 g/l and a boric acid

[0020] A low concentration bath is a sulfate–chloride bath proposed by N.C. Anderson and C. groover, and is a plating bath with the low concentration of a metal ion (JP,57–10191,A). plating conditions -- for example, a nickel chloride -- 35.3 g/l and a nickel sulfate -- 0.75 g/l and a sodium oxide can be made into 25 g/l, and temperature can be made [14.7 g/l and an iron sulfate (II) / 1.2 g/l and a boric acid] into 23 degrees C for 25 g/l and saccharin

[0021] In addition, the plating conditions of a Burns–Warner bath, a Wolf bath, and a low concentration bath are not limited above, and may change plating conditions suitably within limits which can secure the fundamental property of a Burns–Warner bath, a Wolf bath, and a low concentration bath. Moreover, a plating bath is not limited to a Burns–Warner bath, a Wolf bath, and a low concentration bath, and if it is a plating bath which can form the magnetic thin film which consists of a permalloy, all plating baths can be used for it.

[0022] (Mo source of supply) For an addition reason, Mo source of supply is added for Mo ion during a plating bath to a plating bath. As an Mo source of supply, a sodium molybdate, an ammonium molybdate, etc. can be used, for example.

(Organic acid) As an organic acid added during a plating bath, the salt of a hydroxy acid or a hydroxy acid can be used, for example.

[0023] As a hydroxy acid, a tartaric acid, a lactic acid, etc. can be used and the sodium tartrate, a Rochell salt, a sodium lactate, malic-acid sodium, etc. can be used as a salt of a hydroxy acid, for example. From the reason shown below, concentration of the organic acid added to a plating bath can be made into 0.001 or more mol/l, for example, and can be desirably made into 0.01 or more mol/l still more desirably 0.005 or more mol/l.

[0024] Drawing 3 measures the amount of Mo mixing to the inside of the magnetic thin film to Mo ion concentration about each at the time of carrying out organic-acid concentration in mol [0.02 //l.], when an organic acid is not added and organic-acid concentration is made into 0.01 mol/l to calculate the proper value of the addition of an organic acid. The horizontal axis of drawing 3 shows Mo ion concentration under plating bath, and the vertical axis shows the amount of Mo mixing to the inside of a magnetic thin film.

[0025] When not adding an organic acid so that drawing 3 may show, the amount of Mo mixing to the inside of a magnetic thin film will increase rapidly only by Mo ion concentration changing slightly. That is, the rate of change of the amount of Mo mixing to the inside of the magnetic thin film to change of Mo ion concentration is very large. On the other hand, when organic-acid concentration is made into 0.01 mol/l, the rate of change of the amount of Mo mixing in the magnetic thin film to change of Mo ion concentration is small compared with the case where an organic acid is not added.

[0026] Furthermore, when organic-acid concentration is made into 0.02 mol/l, the rate of change of the amount of Mo mixing in the magnetic thin film to change of Mo ion concentration is still smaller. Thus, the rate of change of the amount of Mo mixing in the magnetic thin film to change of Mo ion concentration tends to become small, so that the organic-acid concentration added during the plating bath is high. If the resistivity mentioned later, saturation magnetic flux density Bs, corrosion resistance, etc. are synthetically taken into consideration, it is desirable to make the amount of Mo mixing to the inside of a magnetic thin film into 1 – 5atomic%, and the amount of Mo mixing to the inside of a magnetic thin film can be made into 1 – 5atomic% so stably that organic-acid concentration is high so that drawing 3 may show.

[0027] Therefore, what is necessary is just to make still more desirable desirably 0.005 or more mol/l of organic-acid concentration in mol [0.01 //l. / or more]. It enables this to mix little Mo stably into a magnetic thin film. In addition, organic-acid concentration is not limited to 0.005 or more mol/l, and the organic acid should just be added by the plating bath at least. It may follow, for example, may be good also considering organic-acid concentration as 0.001 or more mol/l, and organic-acid concentration may be still lower. It is possible to mix little Mo into a magnetic thin film, if Mo ion concentration is suitably set up so that the amount of Mo mixing to the inside of a magnetic thin film may serve as a desired value in organic-acid concentration, although change of the amount of Mo mixing in a magnetic thin film [as opposed to / mol / 0.001 //about l, and when low / change of Mo ion concentration] will become large.

[0028] Moreover, the organic-acid concentration under plating bath follows on becoming high, and the amount of Mo mixing to the inside of a magnetic thin film decreases, and Mo ion concentration under plating bath follows on becoming high, and the amount of Mo mixing in a magnetic thin film increases so that drawing 3 may show. From this, the organic-acid concentration to Mo ion concentration is considered to have big influence on the amount of Mo mixing in a magnetic thin film.

[0029] (Organic-acid concentration to Mo ion concentration) Organic-acid concentration to Mo ion concentration can be made into three to 20 times from the reason shown below, for example. Drawing 4 measures the relation between the value which *(ed) organic-acid concentration by Mo ion concentration, and the amount of Mo mixing to the inside of a magnetic thin film to calculate the proper value of the organic-acid concentration to Mo ion concentration under plating bath. The horizontal axis of drawing 4 shows the value which carried out the division of the organic-acid concentration under plating bath by Mo ion concentration under plating bath, and the vertical axis shows the amount of Mo mixing to the inside of a magnetic thin film.

[0030] If the resistivity mentioned later, saturation magnetic flux density Bs, corrosion

resistance, etc. are synthetically taken into consideration, it is desirable to make the amount of Mo mixing to the inside of a magnetic thin film into 1 – 5atomic%, and if organic-acid concentration is set up within the limits of three to 20 times of Mo ion concentration so that drawing 4 may show, the amount of Mo mixing in a magnetic thin film can be made into less than [1–5atomic%].

(Resistivity) The resistivity of the magnetic thin film to the amount of Mo mixing to the inside of a magnetic thin film is explained using drawing 5 . Drawing 5 is a graph which shows the resistivity of the magnetic thin film to the amount of Mo mixing to the inside of a magnetic thin film. The horizontal axis of drawing 5 shows the amount of Mo mixing to the inside of a magnetic thin film, and the vertical axis shows the resistivity of a magnetic thin film.

[0031] Resistivity is changing almost linearly to the amount of Mo mixing in a magnetic thin film, and can obtain the magnetic thin film of desired resistivity by setting up suitably the amount of Mo mixing in a magnetic thin film as mentioned above so that drawing 5 may show. 1 – 5atomic%, then resistivity can be set to about 20 to 100 microomegacm for the amount of Mo mixing to the inside of a magnetic thin film so that drawing 5 may show.

[0032] In addition, since resistivity is proportional to the amount of Mo mixing in a magnetic thin film mostly, although making resistivity of a magnetic thin film high further by making the amount of Mo mixing in a magnetic thin film increase is also considered, when the amount of Mo mixing in a magnetic thin film becomes large so that it may mention later, saturation magnetic flux density Bs will fall, and the corrosion resistance of a magnetic thin film will fall. Therefore, it is desirable to set up suitably the amount of Mo mixing to the inside of a magnetic thin film within limits from which the desired saturation magnetic flux density Bs and desired corrosion resistance are acquired.

[0033] (Saturation magnetic flux density) The relation between the amount of Mo mixing to the inside of a magnetic thin film and the saturation magnetic flux density Bs of a magnetic thin film is explained using drawing 6 . Drawing 6 is a graph which shows the relation between the amount of Mo mixing to the inside of a magnetic thin film, and the saturation magnetic flux density Bs of a magnetic thin film. The horizontal axis of drawing 6 shows the amount of Mo mixing in a magnetic thin film, and the vertical axis shows the saturation magnetic flux density Bs of a magnetic thin film.

[0034] It becomes possible to form the magnetic thin film which has 1 – 5atomic%, then about 0.9 (T) and the high saturation magnetic flux density Bs for the amount of Mo mixing to the inside of a magnetic thin film so that drawing 6 may show. In addition, the amount of Mo mixing to the inside of a magnetic thin film is not necessarily limited to 1 – 5atomic%, and can be suitably set up within limits from which the desired saturation magnetic flux density Bs is obtained.

[0035] (Corrosion resistance) The relation between the amount of Mo mixing to the inside of a magnetic thin film and the corrosion resistance of a magnetic thin film is explained using drawing 7 . Drawing 7 is a graph which shows the relation between the amount of Mo mixing to the inside of a magnetic thin film, and the corrosion resistance of a magnetic thin film. The horizontal axis of drawing 7 shows the amount of Mo mixing in a magnetic thin film, and the vertical axis shows the corrosion resistance of a magnetic thin film. The pitting potential measured by the anode polarization method estimates the corrosion resistance of a magnetic thin film. An anode polarization method is the method of measuring the property of a test piece by measuring the current-voltage characteristic when having arranged by having used the test piece, i.e., a magnetic thin film, as the anode, so that it may counter with a cathode, and applying voltage between anode-cathodes into solutions, such as a sodium chloride. Here, pitting potential means the potential to which current value begins to increase rapidly, after the current-voltage characteristic becomes flat temporarily, when supply voltage is enlarged gradually. Therefore, corrosion resistance will be high, so that pitting potential is high. In addition, the vertical axis of drawing 7 shows the relative pitting potential at the time of setting to 1 pitting potential of the magnetic thin film which consists of the permalloy in which Mo is not mixed.

[0036] 1 – 5atomic% of within the limits, then corrosion resistance can set the amount of Mo mixing to the inside of a magnetic thin film to about 1.0 so that drawing 7 may show. That is, corrosion resistance almost equal to the magnetic thin film which consists of the permalloy in

which Mo is not mixed can be acquired. In addition, the amount of Mo mixing to the inside of a magnetic thin film is not necessarily limited to 1 – 5atomic%, and can be suitably set up within limits from which desired corrosion resistance is acquired.

[0037] Moreover, in within the limits whose amount of Mo mixing to the inside of a magnetic thin film is 1 – 5atomic%, coercive force H_c can obtain magnetic properties with a magnetostriction almost equivalent to the magnetic thin film which consists or less of 2×10 to six, i.e., the permalloy in which Mo is not mixed, below 1 (Oe). Thus, since the amount of Mo mixing in a magnetic thin film can be set as a suitable value by setting the organic-acid concentration under plating bath to Mo ion concentration under plating bath as a suitable value according to this operation gestalt, resistivity is high and can form the magnetic thin film which has good magnetic properties.

[0038] Not only the above-mentioned operation gestalt but various deformation is possible for a [deformation implementation gestalt] this invention. For example, although the above-mentioned operation gestalt explained the case where Mo was mixed in the magnetic thin film which consists of a permalloy to the example, the matter to mix is not limited to Mo, and when mixing W (tungsten), Cr, etc., it can be applied.

[0039]

[Effect of the Invention] Since the amount of Mo mixing in a magnetic thin film can be set as a suitable value by setting the organic-acid concentration under plating bath to Mo ion concentration under plating bath as a suitable value according to this invention the above passage, resistivity is high and can form the magnetic thin film which has good magnetic properties.

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DESCRIPTION OF DRAWINGS

[Brief Description of the Drawings]

[Drawing 1] It is the schematic diagram showing the cell for electroplating.

[Drawing 2] It is the graph which shows the relation between current density and the amount of Fe mixing to the inside of a magnetic thin film.

[Drawing 3] It is the graph which shows the relation between Mo ion concentration under plating bath, and the amount of Mo mixing to the inside of a magnetic thin film.

[Drawing 4] It is the graph which shows the relation between the value which *(ed) organic-acid concentration by Mo ion concentration, and the amount of Mo mixing to the inside of a magnetic thin film.

[Drawing 5] It is the graph which shows the relation between the amount of Mo mixing to the inside of a magnetic thin film, and the resistivity of a magnetic thin film.

[Drawing 6] It is the graph which shows the relation between the amount of Mo mixing to the inside of a magnetic thin film, and the saturation magnetic flux density Bs of a magnetic thin film.

[Drawing 7] It is the graph which shows the relation between the amount of Mo mixing to the inside of a magnetic thin film, and the corrosion resistance of a magnetic thin film.

[Description of Notations]

10 -- Container

12 -- Shield

14 -- Hole

16 -- Anode

18 -- Cathode

20 -- Anode support fixture

22 -- Cathode support fixture

24 -- Plating bath

[Translation done.]